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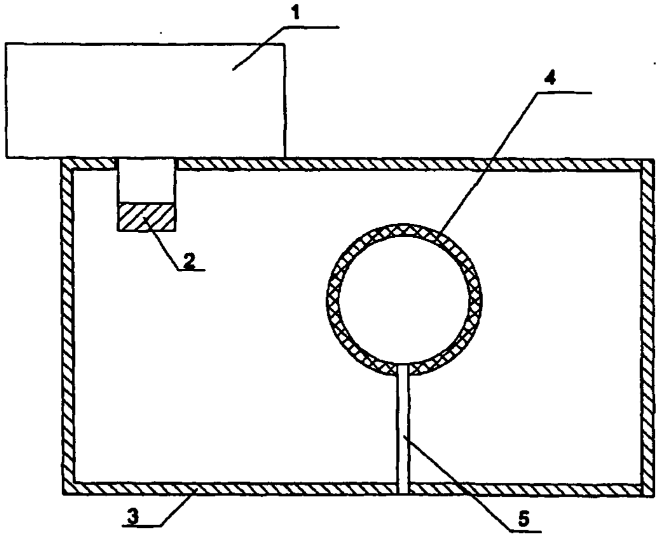
(54) Title: METHOD AND DEVICE FOR STERILIZATION OF MATERIALS BY MEANS OF MICROWAVE RADIATION WITH A HIGH FIELD STRENGTH



(57) Abstract: A method of sterilizing articles by means of microwave radiation comprising increasing the electromagnetic field intensity to a level ensuring microorganisms destruction or sterilization through direct microwave energy absorption by these microorganisms from a high-frequency field. The device consists of a magnetron and a cavity resonator connected by a strong link which results in the magnetron automatically tunes to the resonant frequency of the system: magnetron-resonator-articles being sterilized.

Method and device for sterilization of materials by means of microwave radiation with a high field strength

Field of the invention

The invention relates to the methods and apparatuses for disinfecting and sterilization of various materials using microwave radiation and is intended to be used in simple and compact devices.

Prior art

The necessity to sterilize apparatuses, dressings, dentures and other articles made fully or partially of dielectric materials often arises in medicine. Typically they use autoclave boiling, vapor heating, oven heating. This ensures almost 100% sterilization but takes a long time, while high temperature heating destroys some materials such as plastic.

Other techniques not involving heating such as immersing the materials being sterilized into alcohol, formaldehyde solution, etc take even more time up to several days in some cases.

The third group of methods make use of ultraviolet quartz lamps [Patent US 5547635 , IPC^6 A61L 2/00, appl. 10.11.1993, publ. 20.08.1996], or X-ray or even hard gamma radiation from powerful radioactive sources. Ultraviolet radiation does not ensure the level of sterility required for surgery, since it can only sterilize the surface, while bacteria and spores contained in pores or covered by a thin outer layer nontransparent for ultraviolet radiation remain intact. The X-ray and gamma radiation sterilize the materials only under very high doses amounting to several hundreds thousand or even million roentgens, which requires complex boxes to protect personnel, high radiation levels possibly change mechanical and optical properties of the materials processed.

A method is known where sterilization is being made in gas chambers with high-toxic gas (e.g. Ethylene Oxide) in combination with microwave radiation [Patent US 5209902 , IPC^6 A 61L 002/12, appl. 25.02.1991, publ. 11.05.1993]. The presence of even relatively weak microwave radiation increases the toxic effect of Ethylene Oxide on bacteria.

A microwave radiation has several advantages over the ultraviolet, X-ray and gamma radiation.

It is much more easy to protect personnel from strong microwave radiation than form X-ray and gamma radiation, since a simple several fractions or millimeter thick metal screen is sufficient for full protection form a microwave radiation.

A centimeter band (from 1 to 30 cm) microwave radiation interact only slightly with the majority of dielectric materials such as glass, ceramics, most plastics, fabrics used for dressing, since these materials are almost transparent to a microwave radiation. Thus there is no concealed cavities problem as in the ultraviolet radiation case.

At the same time the centimeter band microwave radiation is strongly absorbed by water, which is a major portion of any microorganism (bacteria are 70-80% composed of water and water makes up some 5-15% of viruses and spores). Thus the microwave radiation action is "selective" compared to ultraviolet. X-ray or gamma radiation. which act uniformly on both microorganisms and on the article being sterilized itself.

Another important property of the microwave radiation in heating up water-containing microorganism is that it acts from inside and not from outside as it is the case in other sterilization methods. Thermal energy is released in the volume of the microorganism itself. Many microorganism and bacterium spores in particular, which have sometimes up to 5 heating resistant protecting shells appear to be unprotected from even small inner heat release, since in this case the protective shell makes the heating easier preventing the heat to transfer from inner volume of the bacteria spores.

However, to generate microwave radiation with a field strength sufficient to destroy microorganisms, power microwave generators are required, and this sterilization method is thus not so widely used.

Invention disclosure

The main goal of this invention is to develop a simple method and device for sterilization of dielectric or partially dielectric materials by means of microwave radiation without using powerful and high-cost generators.

This task is solved in following way. For sterilizing dielectric or partially dielectric materials by means of microwave radiation, microwave source with a power from 100 Wt to 5 kWt is used, the electromagnetic field strength of the source is increased by using a cavity resonator. comprising the steps:

- establishing a link between the microwave source and the resonator,
- placing the articles to be sterilized inside the resonator,
- exciting the electromagnetic oscillation with a frequency corresponding to the resonant frequency of the system: microwave source - resonator - articles sterilized,
- increasing the microwave electromagnetic field intensity to value ensuring destruction of small size biological objects, such as bacteria, spores and viruses, through direct microwave energy absorption by these objects,
- maintaining the microwave field at this level for some time sufficient to sterilize the articles.

In order to exclude the gas breakdown possibility the resonator can be filled with a high breakdown threshold gas (such as freon or elegas) and/or under pressure higher than atmospheric.

When sterilizing the articles, which are bigger than the antinode area of the standing wave in the resonator, the articles are moved inside the resonator during the sterilization process in such a way that all parts of the articles pass consequently through the antinode area.

The method is implemented by a device containing microwave magnetron and a cavity resonator. Inside a cavity resonator there is a radiating element of the microwave magnetron, used as a microwave source. The working frequency of the magnetron differs from the resonant frequency of the resonator by not more than 20 %. Certain mechanism for moving the articles being sterilized and filling the resonator with a high breakdown threshold gases and/or with a pressure higher than atmospheric can be disposed to the resonator.

Comparative analysis have shown that the solution proposed differs from the prototype by the following: The microwave source is used with a power from 100 Wt to 5 kWt. The electromagnetic field strength is increased by using a cavity resonator and establishing a link between the microwave source and the resonator. The articles to be sterilized are placed inside the resonator and the oscillations are excited with a frequency corresponding to the resonant frequency of the system: microwave source - resonator - articles sterilized, increasing the microwave electromagnetic field intensity to value ensuring destruction of small size biological objects.

The essence of the invention is as follows:

To illustrate the method suggested we will make certain estimates. Let us asses the microwave field intensity required to destruct the microorganisms by direct heating of their intrinsic water by a high-frequency electromagnetic field.

Consider the case when a water-containing sample is heated in a conventional microwave oven with a power P = 1 kWt. Let consider the characteristic dimension of the sample, the radius in our case, R = 1 cm, the volume of the sample = 4.2cm^3, water content 100%.

Estimate the energy absorption of this water volume. According to a know formula, the volume power density of the microwave energy loss in a dielectric is directly proportional to square of the electric field strength. the field frequency, dielectric permeability, and the tangent of the dielectric losses angle of the sample studied.

$$W = \varepsilon_0/4\pi\,(E^2 \cdot \varepsilon \cdot \omega \cdot \mathrm{tg}\delta) \qquad (1),$$

where

W is the volume power density of the microwave energy loss,

E is the microwave field strength (average over the period),

ε is the relative dielectric permeability of the sample,

$\varepsilon_0 = 8.85 \cdot 10^{-12}$ F/m is the vacuum absolute dielectric permeability,

ω is the microwave radiation angular frequency,

$\mathrm{tg}\delta$ is the dielectric losses tangent for the sample substance.

For the microwave oven case the power being radiated is about $P =$ 1kWt corresponding to the field intensity $E = 200 - 400$ V/cm (depending on the oven volume). Substituting $E = 300$ V/cm, $\varepsilon = 80$ (for the water under room temperature), $\omega/2\pi = 2.45$ GHz, $\mathrm{tg}\delta = 0.1$ (for the frequency 2.45 GHz and the water under room temperature), we get $W = 80$ Wt/cm^3. This means that each gram of water when placed in the microwave field with a frequency 2.45 GHz and strength $E = 300$ V/cm absorbs the power 80 Wt, which heats up the water.

Estimate the heating rate of the water sample with the radius of 1 cm. First estimate the energy losses. The energy is lost due to heat transfer to the outer air and due to thermal radiation. The heat transfer losses are proportional to the surface area of the sample, temperature gradient and thermal conductivity coefficient

$$q = \lambda \cdot S \cdot \mathrm{grad}T \qquad (2),$$

where

q is the heat losses power from the sample surface,

S is the sample surface area.

gradT is the temperature gradient. In our case it may be estimated as the difference between the outer air temperature and the water sample temperature divided by a characteristic dimension of the task, i.e. the sample radius. This is permissible for a rough estimate.

For air $\lambda = 0.023$ Wt/m·K. Neglecting the thermal resistance of the water-air border we get the heat transfer losses $q = 0.05$ Wt for a water-air temperature difference about 80 K. If a water sample is placed on a dielectric surface these losses will be slightly higher (due to higher heat conductivity) but in any case not exceeding 0.5 Wt.

The thermal radiation losses for the water temperature $T = 373$K (water boiling temperature) do not exceed:

$$Q = S \cdot \sigma \cdot T^4 \qquad (3),$$

where·

Q is the thermal radiation energy loss power,

S is the surface area

T is the absolute temperature of the surface

$\sigma = 5.7 \cdot 10^{-8}$ Wt/m^{2}K^4 is the thermal radiation power of the absolutely black body, giving Q=1.4 Wt.

Thus the total of losses do not exceed in any case 2 Wt and consequently much less (approximately two orders of magnitude less) than the microwave energy fed to the sample. The energy fed for our sample case is about 340 Wt and the losses can be neglected in the first order approximation.

In this case to heat a sample with a mass of 4.2 gram by 80 degrees (i.e. from a room temperature to a boiling temperature) will require the energy of 1400 J and the heating time will be 5 sec (the water heat capacity assumed to be 4.2 J/g).

Consider the same example for the case when the water sample dimension is decreased from 1 cm to 1 mcm (i.e. by a factor of 10000), which corresponds to the average bacterium size. In this case the whole system still remains macroscopic (i.e. comprising at least several million molecules and the system size is much bigger than the average free path of the air molecule) all the above presented formulas remain valid.

Since the losses are proportional to the surface area (and in case of heat transfer they are proportional to the temperature gradient which also increases by 10000 times) and the energy absorbed is proportional to the volume, the energy losses due to heat transfer and thermal radiation substantially exceed in this case the microwave energy absorbed.

For a case when a single bacterium is place in the air in the microwave field with an intensity of a conventional microwave oven, the energy absorbed is $3 \cdot 10^{-10}$ Wt. The energy lost due to heat transfer when sample is heated by 80 degrees is $5 \cdot 10^{-6}$ Wt and the energy lost due to thermal radiation is $1 \cdot 10^{-8}$ Wt.

Thus the energy lost due to heat transfer to the air molecules exceeds by four orders of magnitude the energy absorbed (the thermal radiation losses can be neglected).

This makes it impossible to heat up an isolated bacterium placed in the microwave field with an intensity of a conventional microwave oven. This means bacteria placed in such a field remain intact (provided there is no additional factors such toxic gas, as in the patent US5209902).

In order to heat a bacterium in the microwave field it is necessary to increase the field strength, according to the equation (1) by at least 100 times (all other values remain the same). Such a field intensity makes the energy absorbed and the energy lost due to heat transfer and thermal radiation to be approximately equal for a 1mcm bacterium, making it possible to strongly heat up a single bacterium.

So, for an efficient sterilization by direct heating of the microorganism with the microwave radiation, the microwave field strength for 2.45 GHz frequency must be at least 30 kV/cm.

This is not so difficult to increase the microwave field intensity to a level required. A cavity resonator of the microwave band with a sufficiently high quality factor can be used.

For typical cylinder resonators excited at the TM010 mode the field strength at the center of the resonator is, according to [McDonald. Microwave breakdown is gases]

$$E_0 = \sqrt{P_0 Q / \eta \omega_0}, \qquad (4)$$

where

P_0 is the magnetron microwave radiation power,

Q is the resonator quality factor,

ω_0 is the microwave radiation angular frequency,

$$\eta = 0.27 \cdot \varepsilon_0 \cdot V \qquad (5)$$

where

$\varepsilon_0 = 8.85 \cdot 10^{-12}$ F/m is the vacuum absolute dielectric permeability,

V is the resonator volume, chosen to be $V \sim \lambda^3$,

where λ is the length of the microwave in free space.

For example, when P_0 = 1kWt, $\omega_0/2\pi$ = 2.45 GHz, V = 1l, Q = 10^3 the electric field strength will be $E_0 \sim 160$ kV/cm.

This is about 500 times higher than for the case of heating in a conventional microwave oven. In such a strong electromagnetic field the energy absorbed by a bacterium considerably (about 20 times) exceeds the upper estimate of thermal losses (bacterium size assumed to be around 1 mcm).

Using the equation (1) we get that the energy absorbed by a single bacterium with a characteristic size 1 mcm is $\sim 8 \cdot 10^{-5}$ Wt. Neglecting the thermal losses in this case it is possible to asses the bacterium heating rate. This rate appears to be very high due to the fact that when a liner dimension changes by a factor of 10000 the volume changes by a factor of 10^{12}, while the power input decreased by a factor of only $340/8 \cdot 10^{-5} \sim 4 \cdot 10^6$.

Thus the heating rate of a bacterium in strong field created inside the resonator exceed by a factor of than a million the heating rate of a water sample with 1cm radius inside a conventional microwave oven. To heat a bacterium to the boiling temperature with a field strength inside the resonator requires about 1 mcsec.

The very short time needed to heat up a bacterium in a high intensity microwave field has another advantage. Since the process of heat transfer requires certain time to develop , the real energy losses due to heat transfer will be considerably less during such a short time compared to those for the stationary equilibrium heating. This allows to reach appropriate balance between the energy absorption and radiation by a bacterium at lower microwave field intensity.

The field intensity required for bacterium heating appears to be rather high being of the order or several times higher than the breakdown threshold of the air at atmospheric pressure (~30 kV/cm for a microwave band). This problem is solved by filling up the resonator by a certain high breakdown threshold gas (e.g. elegas or freon have the breakdown threshold at atmospheric pressure at least tenfold higher than the air).

Using cavity resonator for microwave breakdown of gases is known for several decades. Usually complex systems are used, which contain a sufficiently powerful microwave generator (typically a magnetron type with several kWt power), waveguide transfer lines, microwave loading, branching waveguides to transfer a portion of the microwave generator radiated power to a resonator, precision frequency tuning systems, stabilizing devices for the generator frequency and the resonator temperature, power control system, etc.

In such installation the resonator is very weakly linked with the magnetron generator by means of long chain containing coordinating and control devices. Thus such installations appear to be rather inconvenient and consumes considerable electric power. At the same time, only a small fraction of the total power consumed is used to excite microwave oscillations inside the resonator. Meanwhile all the above-mentioned devices and control systems are needed only for "purity of experiments" and can be abandoned in practice when a cavity resonator is used. This requires establishing a strong link between the magnetron and the cavity resonator, which is implemented in this invention.

A cavity resonator can be utilized to tune and stabilize a magnetron frequency. In this case additional resonance states appear in the magnetron resonance system regardless of the way the magnetron anode block is coupled with the resonator. [D.V. Samsonov, Osnovu rascheta i konstruirovania magnetronov, Moscow, Sovetskoye Radio, 1974, pp 167-194].

According to the "minimum dissipation" principle, the "magnetron-resonator" system oscillations are excited at the frequency where the ratio of the energy conserved to the total loss energy over a period reaches its maximum. This is a maximum quality factor frequency. Since the magnetron resonance system quality factor (~100 usually) is much less than that of the cavity resonator (~ 1000 – 10000 usually), the oscillations are excited at the resonant frequency of the magnetron-resonator system. Naturally this frequency should be fairly "close" to the magnetron working frequency. The degree of closeness depends on the magnetron type and how strongly it is linked with the resonator.

A strong link can be established for example if the magnetron radiating element is placed inside the resonator.

All these condition being met, the energy starts to build up within the cavity resonator and a standing wave is being formed with the maxima and minima of electric field spaced depending on the specific cavity resonator type (cylindrical. coaxial. rectangular. etc.) and the type of the oscillation excited.

In this situation, the electric field strength in the standing wave antinodes is much higher than the magnetron electric field in a free space. For high quality factor resonators such an intensity increase can be very high, up to 100 times and more.

Usually when a cavity resonator is used to achieve a microwave breakdown, a gas discharge which forms when intensity reaches a breakdown level, spreads over the whole resonator volume. This results in the sharp decrease of the field intensity inside the resonator due microwave energy dissipation by the gas discharge formed. However, if a relatively low power microwave generator is used and/or the resonator is filled up with the low breakdown threshold gas, the field intensity appears to be high enough to destroy the microorganisms but not so high to reach the breakdown, which makes it possible to use such device for sterilization.

If a strong link is established between the microwave source and the cavity resonator, the system comprising the magnetron, the cavity resonator and the article sterilized will become a single resonator with its own resonant frequency, quality factor and the standing wave pattern. The standing wave patterns for resonator with simple shapes such as cylinder, rectangular and coaxial are described elsewhere. But since the strong link distorts the field patter even in these simple cases preliminary research is needed for the standing wave pattern. This can be carried out by simple microwave measuring devices (e.g. by the microwave probe). After identifying the standing wave antinodes the sterilized article is placed in one of the antinodes. Then the oscillations are excited at the resonant frequency of the system comprising the microwave source, resonator, sterilized article, thus increasing the field intensity in the sterilized article location to a level ensuring the destruction of the microorganisms both at the surface and inside the sample.

If the sterilized article is moved within the resonator, there is no need to investigate the standing wave pattern inside the resonator, since it is known that the standing wave antinodes occupy at least half a resonator volume. In this case, since mechanically moving the article is much slower than the magnetron frequency set time, the magnetron microwave generator will have enough time to follow the proper resonant frequency of the system (the resonant frequency depending upon the article location inside the resonator). Thus it maintains the high field intensity at any time.

The device contains the magnetron with a relatively low power (100 Wt to 5 kWt). The radiating element is disposed inside the resonator. The working frequency of the magnetron should differ from that of the cavity resonator by not more than 20%.

The sterilized article when placed inside the resonator changes the proper resonant frequency of the system. But since the dielectric losses of the majority of the dielectric materials used in medicine (such as polyethylene, glass, ceramics. dressing materials) are rather low in a microwave band, the quality factor of the cavity resonator remains essentially unchanged. In this case the magnetron tunes automatically to the resonant frequency of the system comprising the magnetron. the resonator, and the articles being sterilized. When there is a big difference between the fundamental working frequency of the magnetron and the system resonant frequency exceeding several percent, the magnetron oscillation can excite at the parasitic oscillation mode. This can decrease the effective radiation power. But since the magnetron works at this mode very short time (several seconds), there is no overheating and failure of the magentron.

Unfortunately it is quite difficult to use the method suggested to sterilize the material containing open metal parts. This because there will be a breakdown in the gas filling the resonator by a discharge, which will form in the field heterogeneity area in the metal part vicinity. This does not allow to reach the field strength required for efficient sterilization.

The sterilization of the articles containing metal parts covered by a dielectric material is possible.

BRIEF DESCRIPTION OF THE DRAWINGS

The apparatus work is illustrated by the Figure 1.

The device contains a microwave magnetron 1, a radiating element 2 placed inside a cavity resonator 3. and an article being sterilized 4 also placed inside a cavity resonator. Optionally there can be an auxiliary element 5 for mechanical movement of the sterilized article within the resonator.

The apparatus works in the following way

After the voltage is supplied to the magnetron 1 a frequency corresponding to the resonant frequency of the magnetron 1- resonator 3 - sterilized article 4 system is set within a very short time. Then energy builds up inside the resonator 3 and within $\sim 10^{-6}$ sec reaches a necessary level, after the microorganisms in the article and on its surface are destroyed due to the microwave energy absorption. If it is necessary to sterilize bulky articles, the device is complemented by an auxiliary element 5 to move the sterilized article through the standing wave antinodes. For proper sterilization the article is subjected to microwaves at a high level for several seconds.

EXAMPLE OF USE

We used a magnetron with a power supply from a conventional microwave oven with a power P = 800 Wt and the frequency $f = \omega_0/2\pi$ = 2.45 GHz. It has been thus possible to use resonator made of copper with a volume V = 0.7 l for the wavelength λ = 12 cm. The resonator quality factor was about $Q \sim 10^3$.

The article for sterilization was dried culture of a sporogenous bacterium, the so-called "Hay bacillus", which is well known for its high vitality and which is quite difficult to sterilize by conventional methods (the size of a single bacterium is ~ 2 mcm). The material was place on a microscope slide with dimension 5 by 2 cm and the thickness 0.2 cm. Before the sample is placed in the resonator both single bacteria and colonies of bacteria can be observed on the slide. After keeping the slide in the resonator for 3 second the slide was inspected under the microscope. There were no traces of bacteria, colonies or spores. At the same time the slide remained virtually unheated.

The method suggested allows to use a device which is very compact and simple. There is no need to use any tracing or control systems. There is no problem to tune the magnetron and the resonator onto the same frequency and thus there is no time or thermal instability due to imprecise frequency tuning.

This makes it possible to use the apparatus in medicine for quick sterilization of dielectric or partially dielectric materials. The device itself being very compact and simple makes it possible to use it in the field for dressing and surgery material sterilization from such microorganisms as bacteria and bacterial spores.

It is also possible to use damp materials which are first dried by a microwave field at low field intensity (as in a conventional oven). After water vaporizes the resonator transits to high quality factor state, the sterilization is then takes place at a high field intensity.

CLAIMS

1. A method of sterilizing dielectric or partially dielectric materials by means of a microwave source with a power from 100 Wt to 5 kWt by using a cavity resonator for increasing the electromagnetic field strength, comprising the steps:
   - establishing a link between the microwave source and the resonator,
   - placing the articles to be sterilized inside the resonator,
   - exciting the electromagnetic oscillations with a frequency corresponding to the resonant frequency of the system: microwave source - resonator - articles sterilized,
   - increasing the microwave field strength to the value ensuring destruction of small size biological objects, such as bacteria, spores and viruses, through direct microwave energy absorption by these objects,
   - maintaining the microwave field at this level for some time sufficient to sterilize the articles.

2. The method as claimed in 1, wherein during the time when the microwave field intensity is maintained at the level ensuring the small size biological object destruction, the articles being sterilized are moved inside the resonator.

3. The method as claimed in 1 or 2, wherein during the time when the microwave field strength is maintained at the level ensuring the small size biological object destruction, the resonator is filled with a gas or gas mix with a high breakdown threshold, such as freon or elegas, and/or under pressure higher than atmospheric.

4. The method as claimed in 1 to 3, wherein when damp articles are sterilized a low microwave field strength is first maintained inside the resonator and only after the articles become completely dry the

microwave field intensity level is increased to destroy small size biological objects.

5. The apparatus to implement the method claimed in 1 to 4, containing a cavity resonator wherein a radiating element of a microwave magnetron which is used as a microwave radiation source is placed inside the resonator, and a working frequency of the magnetron differing from a proper frequency of the cavity resonator by no more than 20%.

6. The apparatus as claimed in 5, wherein the cavity resonator is complemented by a system to move the article being sterilized inside the resonator.

7. The apparatus as claimed in 5 or 6, wherein the cavity resonator is complemented by a device to fill the resonator with a gas with a high breakdown threshold and/or under pressure higher than atmospheric.

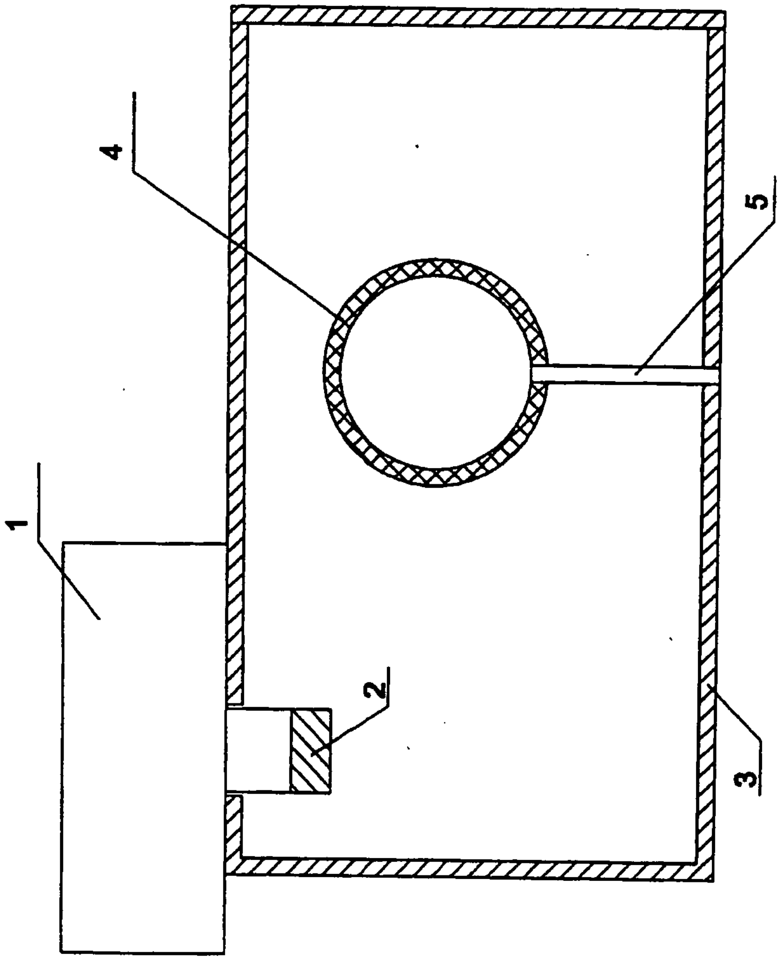


Fig.1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU 00/00274

A. CLASSIFICATION OF SUBJECT MATTER

A61L 2/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
A61L 2/00,2/08, 2/12, B65F 7/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched:

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	US 5209902 A (IAN P. MATTHEWS et al) 11 May 1993 (11.05.93)	1-7
A	RU 2098134 C1 (AKTSIONERNOE OBSCHESTVO "TITAN") 10 December 1997 (10.12.97)	1-7
A	RU 2036663 C1 (TROFIMOV VLADISLAV IVANOVICH et al) 9 June 1995 (09.06.95)	1-7
A	RU 2130319 C1 (SARATOVSKY GOSUDARSTVENNY TEKHNICHESKY UNIVERSITET) 20 May 1999 (20.05.99)	1-7

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex

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